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EFFECT OF STRESS ON RECRYSTALLIZATION OF LOW CARBON STEEL

Richard E. Munly

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by

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Lieutenant, United States Navy

Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE
IN
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ABSTRACT

This thesis reports a study to determine the effect of elastic stress on the recrystallization of an open hearth low carbon steel cold worked 50% from the as received hot rolled condition. A standard lever arm creep unit was used to apply a tensile load to tapered tensile specimens which were heated in a salt bath. Rockwell B hardness measurements were used to follow the course of recrystallization. No evidence was found that stress influenced the recrystallization rate.

There was evidence that plastic strain had an accelerating effect on the recrystallization but no quantitative data were obtained.

The recrystallization activation energy of the steel employed in the tests was determined to be 108,000 cal/gm-mol.

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INTRODUCTION

In the majority of design applications, metals are stressed below the elastic limit. It is also true that in many of today's engineering problems, weight limitations and high temperatures loom large in importance. Advantage is often taken of the increased strength obtainable by cold working, but a cold worked metal is subject to recrystallization and softening at a temperature which depends on the material. It is therefore important to know if recrystallization is affected by stress in severe design applications.

Although strain comes as a result of stress, plastic strain and stress may be thought of as separate factors which might affect the recrystallization process. Since in most applications metals are stressed below the plastic region it was the purpose of this investigation to determine the effect of stress in the elastic region on the recrystallization relation.

In a creep rupture study of Monel by Grant and Bucklin,[1*] it was determined that recrystallization occurred at lower temperatures and in shorter times than in static tests (tests under no load). It is not possible to tell from this study whether a similar effect would be observed with a stress in the elastic region.

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[*] Superscripts refer to the bibliography, p. 32.

It is known however that stress has an effect on the martensite transformation. Chang and Read [2] showed that in a gold-cadmium alloy of 47.5 per cent cadmium, a stress corresponding to bending a 1/8 inch square cross section specimen over a span of 1.5 inches to give a displacement of 0.1 inch, produced an increase in the temperature of the beginning of martensite transformation and a decrease in the temperature at which the reverse transformation began.

Kehl and Bhattacharyya [3] in an investigation of the influence of tensile stress on the isothermal decomposition of autenite to ferrite and pearlite reported that applied stress markedly affected both the beginning and ending times of transformation and accelerated the rate of transformation.

In view of the reported effect of stress on transformations and the effect of strain on recrystallization, it seemed reasonable to suspect that stress in the elastic region would affect recrystallization. Since for practical design considerations such an effect would be of importance, it was decided to investigate whether or not stress does affect recrystallization behavior.

DISCUSSION OF RECRYSTALLIZATION

There are three processes which occur in annealing a cold worked material: recovery, recrystallization and grain growth. Recovery may be defined as a change in the properties unaccompanied by any microstructural changes. Grain growth follows recrystallization and is continuation of growth of recrystallized grains. Recrystallization is discussed more fully below.

Metals which are cold worked consist of a matrix of strained crystals which are metastable. Recrystallization is a process by which new strain-free crystals nucleate and grow consuming the cold worked matrix. Recrystallization is not an instantaneous process, but rather its rate of progress follows an exponential law.

A summary of qualitative laws of recrystallization has been presented by Burke and Turnball.[4]

1. a minimum deformation is necessary to cause recrystallization.
2. the smaller the degree of deformation the higher is the temperature required to cause recrystallization.
3. increasing the annealing time decreases the temperature necessary for recrystallization.
4. the final grain size depends chiefly upon the degree of deformation, and to a lesser extent on the annealing temperature, being smaller the greater the degree of deformation and the lower the annealing temperature.
5. the larger the original grain size, the greater is the amount of cold deformation required to give equivalent recrystallization temperature and time.
6. the amount of cold work required to give equivalent deformation hardening increases with increasing temperature of working.
7. continued heating after recrystallization is complete causes the grain size to increase.

The classical Arrhenius equation for activated processes is known to apply to recrystallization. It is discussed in any Physical Chemistry text such as Prutton and Maron.[5] It may be stated as follows: $r = r_o \exp(-Q/RT)$ where r is the recrystallization rate; r_o is a constant; T is the absolute temperature, R is the universal gas constant, and Q is the activation energy. According to the concept of activation, reactants do not pass directly to products but must first acquire sufficient energy to climb over an activation energy barrier.

The applicability of this law to the recrystallization of metals has been pointed out by Mehl[6] who states: "Whether the times for beginning recrystallization, or half or full recrystallization, or rates of nucleation and growth are employed, it is always observed that the rates vary exponentially with the reciprocal of the absolute temperature; that is, a plot of the logarithm of the rate versus the reciprocal of the absolute temperature is a straight line. The slope of this curve which is the temperature coefficient of the process, furnishes the energy of recrystallization."

A formal theory of recrystallization kinetics has been evolved in terms of nucleation frequency, $(\dot{N})$, and linear rate of growth, (G), by Johnson and Mehl[7] and Avrami.[8] It is possible to distinguish activation energy of nucleation, $(Q_{\dot{N}})$, from activation energy of growth, (Q_G), by measuring the nucleation rate and growth rate separately. Burke and Turnbull[4] have presented activation energies (Q), from several sources, obtained by plotting the logarithm of the reciprocal of time required for a constant fraction of the

material to recrystallize versus the reciprocal of absolute temperature. Although Q is not necessarily equal to Q_G because it is partly determined by $Q_{\dot{N}}$, still for the cases considered they point out that Q proved independent of the fraction of material recrystallized and they suggest that the most reasonable interpretation is that $Q \doteq Q_G \doteq Q_{\dot{N}}$.

The activation energy may not be a constant value for all temperatures. The recrystallization activation energy for aluminum has been reported as 34.0 kilocalories per gram atom by Kornfeld and Pawlow[9] and as 59.0 kilocalories per gram atom by Anderson and Mehl.[10] The difference may have been due to factors such as purity, history, or mode of deformation. It is also possible that different activation energies are obtained for different temperature ranges. Burke and Turnbull[4] have replotted the data from the two investigations above and have shown that a continuous curve could be drawn through the data on a plot of log of growth rate (G) versus reciprocal of absolute temperature.

Using the equation $r = r_o \exp(-Q/RT)$ as the basic kinetic equation of recrystallization under static conditions, it is reasonable to ask in what way stress would enter the equation, or to find the function of stress in the following equation

$$r = r_o \exp(-Q/RT)\ f\ (s)$$

It was the aim to perform static recrystallization studies to obtain the activation energy and then to study the recrystallization process under stress.

TEST MATERIAL

The material chosen for this study was an open hearth low carbon steel supplied by the Kaiser Steel Co., Fontana, California. The analysis as supplied with the material is shown in Table I.

TABLE I

ANALYSIS OF MATERIAL

Element	Percent	Element	Percent
C	0.06	Al	0.012
Mn	0.34	Co	0.035
P	0.010	Mo	0.005
S	0.030	Cr	0.010
Si	0.010	Ni	0.02
Cu	0.08	Fe	remainder

The material was received as hot rolled sheet of an initial thickness of 0.128 inches. The sheet was sheared into one inch strips to facilitate rolling and fabrication of specimens.

It has been shown by Channon and Walker[11] in a study on cartridge brass that the recrystallization activation energy decreases with increasing deformation. To avoid the possibility of such an occurrance, only a single percent deformation was used. The material for all specimens was rolled to 50% reduction. This value of reduction was chosen as the maximum practicable because concern must be given to minimum specimen thickness in taking hardness readings.

The effect of number of passes through the roller to arrive at 50% reduction was investigated and it was found that the hardness was the same whether many passes or few passes were made.

After rolling, some strips were sheared into specimens approximately one inch by one-half inch for static recrystallization study. Other strips were fabricated into specimens for stress study and are described later.

USE OF HARDNESS READINGS

The progress of recrystallization in a cold worked metal can be followed by observing the change in one of several physical properties such as hardness, tensile strength, shift in the preferred orientation of the crystallites, or magnetic induction. A more fundamental method of following recrystallization is by microstructural observations. However, this method is both tedious and leads to considerable error due to the uncertainty in distinguishing between strained and recrystallized grains and due to the relatively small area covered unless a statistical evaluation is made.

In discussing the recrystallization of iron, Mehl[6] states that the Brinnel hardness decreases rapidly upon recrystallization and only a very slight decrease as a result of recovery occurs before this.

Cook and Richards[12] used diamond pyramid hardness readings to follow recrystallization in copper. A microscopic study to obtain percent recrystallization by visual estimate was compared with hardness readings. They found the linear relationship

$$X = 0.1 + .9 \frac{H_o - H_x}{H_o - H_1}$$

where H_x is the hardness when fraction X is recrystallized, H_o is the maximum initial hardness, and H_1 is the final end point hardness.

In analyzing the work of Cook and Richards, Thorley[13] showed

that it is not necessary to know the exact correlation between hardness and percent recrystallization in order to find activation energy. He stated, "While the overall correlation between H_x and X may be

$$0.1 + .9 \frac{H_o - H_x}{H_o - H_1}$$

the hardness responds to changing X in the steep part of the annealing curves as though $H_x = H_o(1 - X)$."

It is clear from this approximation that a hardness halfway between initial and final values would correspond to a condition of close to 50% recrystallization. It has been previously stated that for any arbitrary constant value of X and for a particular cold worked state, the logarithm of time is a linear function of the reciprocal of the absolute annealing temperature. Interpolated values of half hardness time from annealing curves may be plotted versus the reciprocal of the absolute temperature and the slope of the straight line resulting is a measure of the activation energy of recrystallization. Fifty percent is a good choice for X because it corresponds to the steepest portion of the annealing curve and thus an error in percent recrystallization makes the smallest difference in half hardness time.

It was decided that hardness readings would be used to follow recrystallization in this study and consequently various hardness scales were examined in order to choose the most suitable. The Rockwell B scale was finally selected because it was appropriate for the hardness of the material involved and the numerical range of the

scale was relatively great, thus contributing a greater sensitivity to the readings.

In discussing Rockwell hardness testing the ASTM[14] states that the thickness of the piece tested should be such that no bulge showing the load effect appears on the surface opposite the impression. They also state that tests on annealed iron of Rockwell B 35 hardness showed that the readings were not affected down to a minimum thickness of 0.45 inches, although the impressions showed through on material 0.06 inch or under in thickness.

In view of the above and because of the fact that no bulges showed through on any specimens tested, it was felt that the specimens were sufficiently thick (approximately 0.064 in.) to test with the Rockwell B scale.

In order to confirm that there is a correlation between hardness measurements and recrystallization, a series of photomicrographs were prepared using specimens heated at 525°C. These photomicrographs are shown in Figs. 1 through 8 and show clearly that recrystallization was progressing as the hardness decreased.

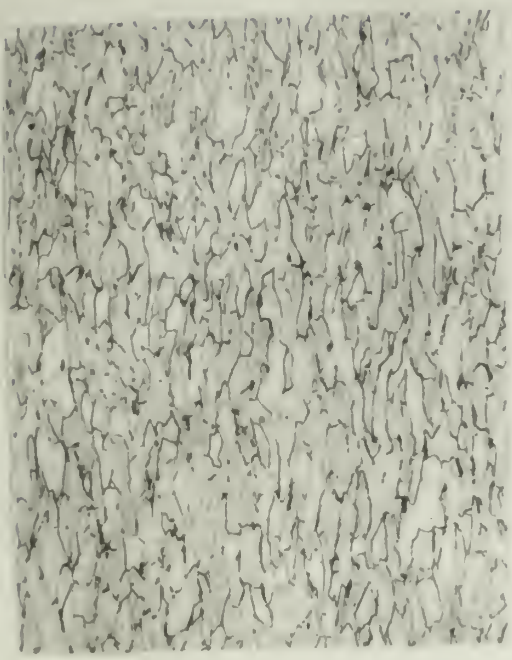


Fig. 1
As Rolled Condition
(Rockwell B 97)

Fig. 2
At 525°C for 6 min.
(Rockwell B 96)

Fig. 3
At 525°C for 2 hrs.
(Rockwell B 93)

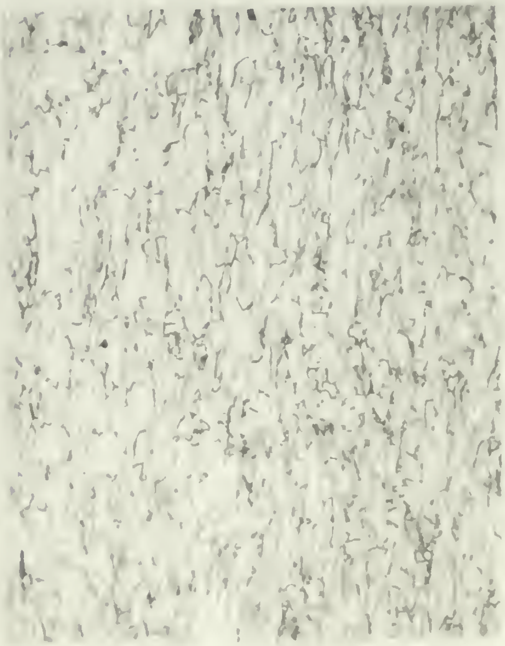


Fig. 4
At 525°C for 5 hrs. 50 min.
(Rockwell B 90)

Fig. 5
At 525°C for 11 hrs.
(Rockwell B 84.5)

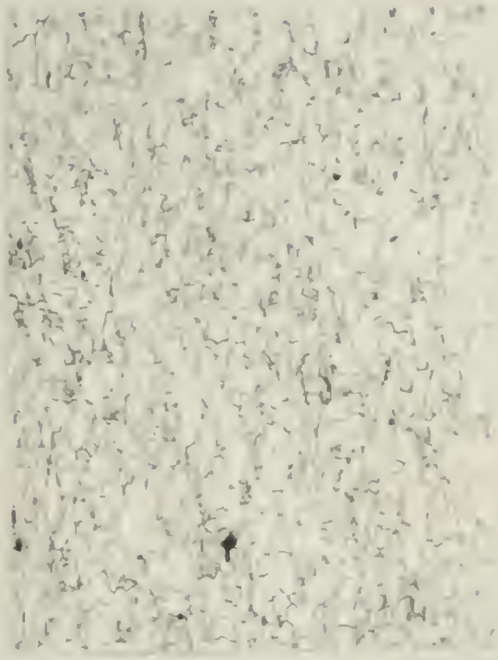


Fig. 6
At 525°C for 17 hrs. 45 min.
(Rockwell B 78)

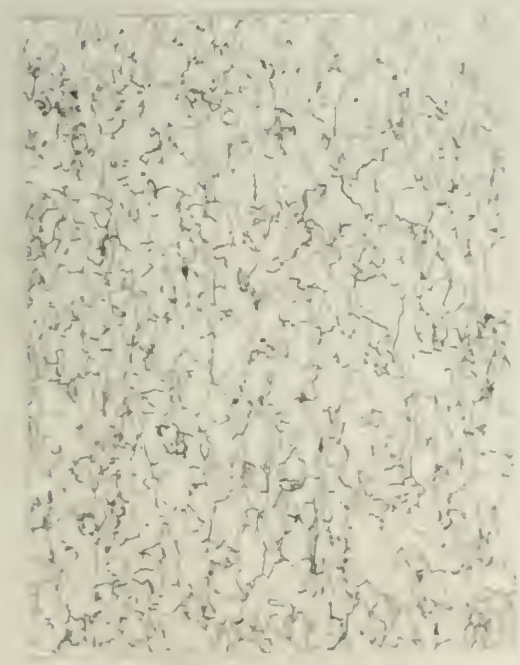


Fig. 7
At 525°C for 24 hrs.
(Rockwell B 64.5)

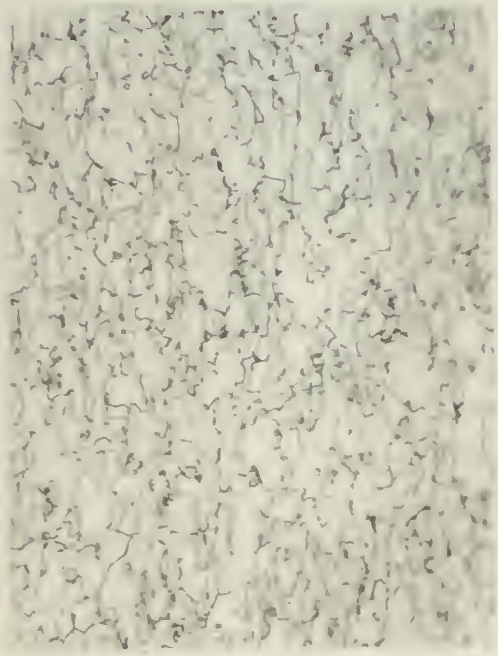


Fig. 8
At 525°C for 1 week
(Rockwell B 65)

STATIC RECRYSTALLIZATION STUDY

For the static recrystallization study both muffle air furnaces and a salt bath were used. The salt bath was used at the higher temperatures where the times were shorter in order to decrease the time for the specimens to come up to temperature. The salt used was Houghton Liquid Heat No. 235. It is a eutectic composition of lithium, sodium, and potassium carbonates and can be used in the range 800 to 1700°F.

A microscopic examination of specimens after test showed no surface effect due to the salt which would otherwise affect the hardness readings.

Four or more hardness readings were taken on each specimen after polishing off the slight adherent oxide, and an average value was used. No difference was noted in hardness readings when the oxide was not removed from the specimens. This was undoubtedly due to the breaking through the scale by the minor load of the Rockwell hardness tester. However, the scale was removed for all specimens used.

Sigmoidal annealing curves for temperatures 525°C, 552°C, 580°C, and 605°C are shown in Figs. 9 through 12. Interpolated values for the time of half hardness were taken from these curves and are plotted versus the reciprocal of absolute temperature in Fig. 13.

Appendix I shows the calculation to obtain the activation energy. A value of 108,000 cal/gm-mol was obtained. This compares closely with a value of 110,000 cal/gm-mol obtained by Burke and

Chandler[15] for a Westinghouse puron deoxidized alpha iron and with the values of 90,000 cal/gm-mol to 113,000 cal/gm-mol obtained by Miller[16] for various commercial carbon steels. These values obtain- by Burke and Chandler and by Miller are for the activation energy of grain growth, but as was previously stated $Q \doteq Q_G$.

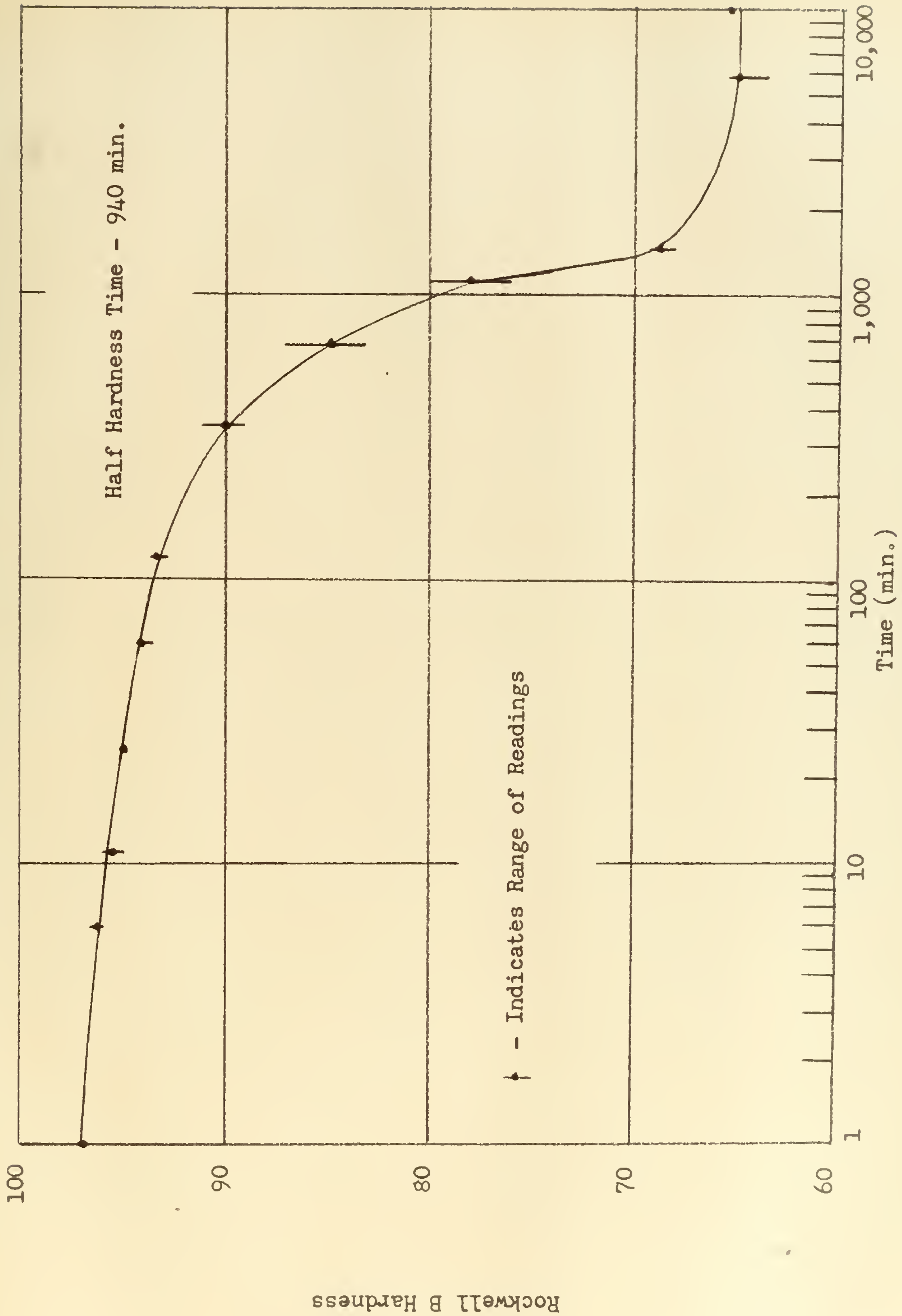


Fig. 9
Annealing Curve (525°C)

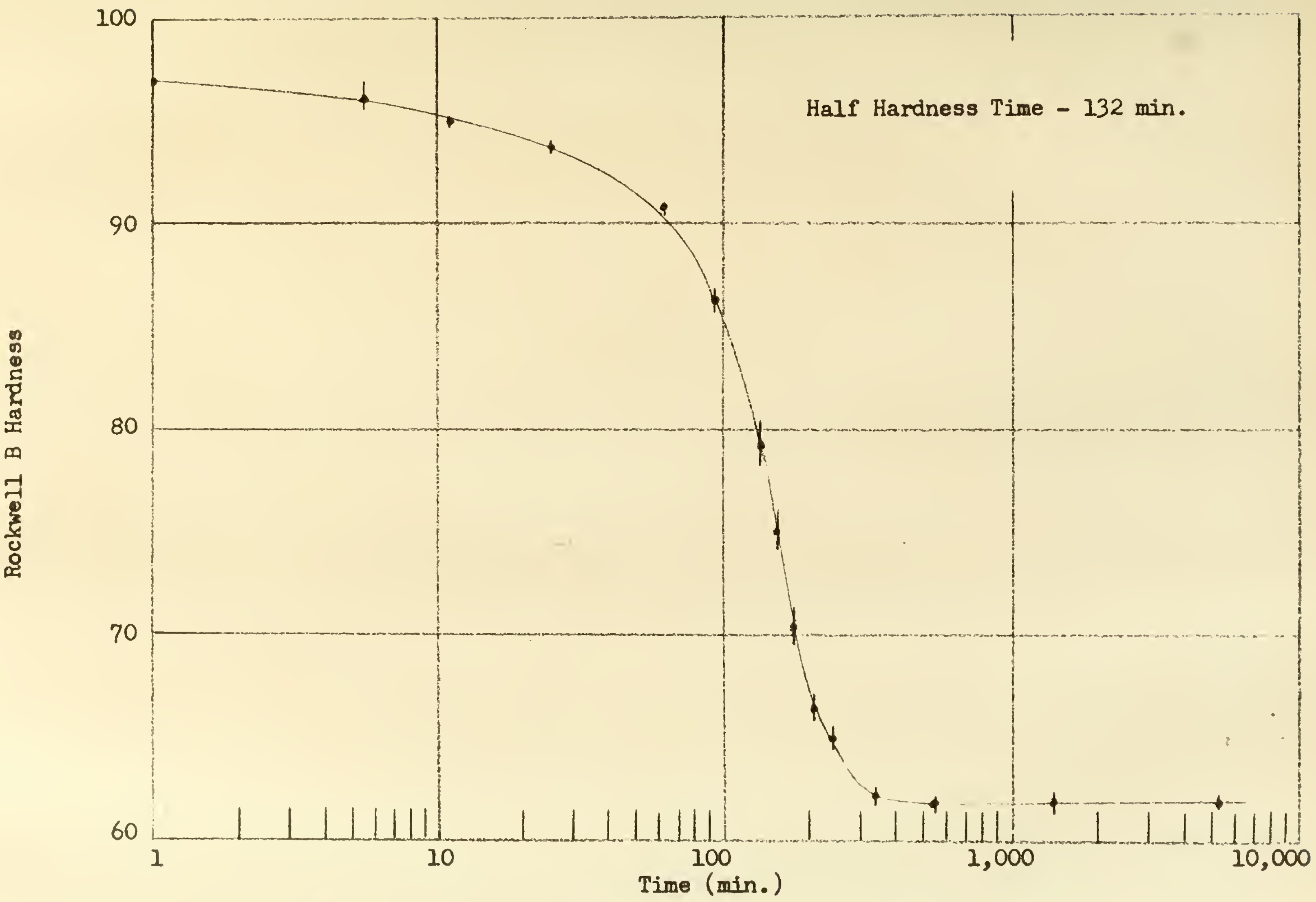


Fig. 10
Annealing Curve (552°C)

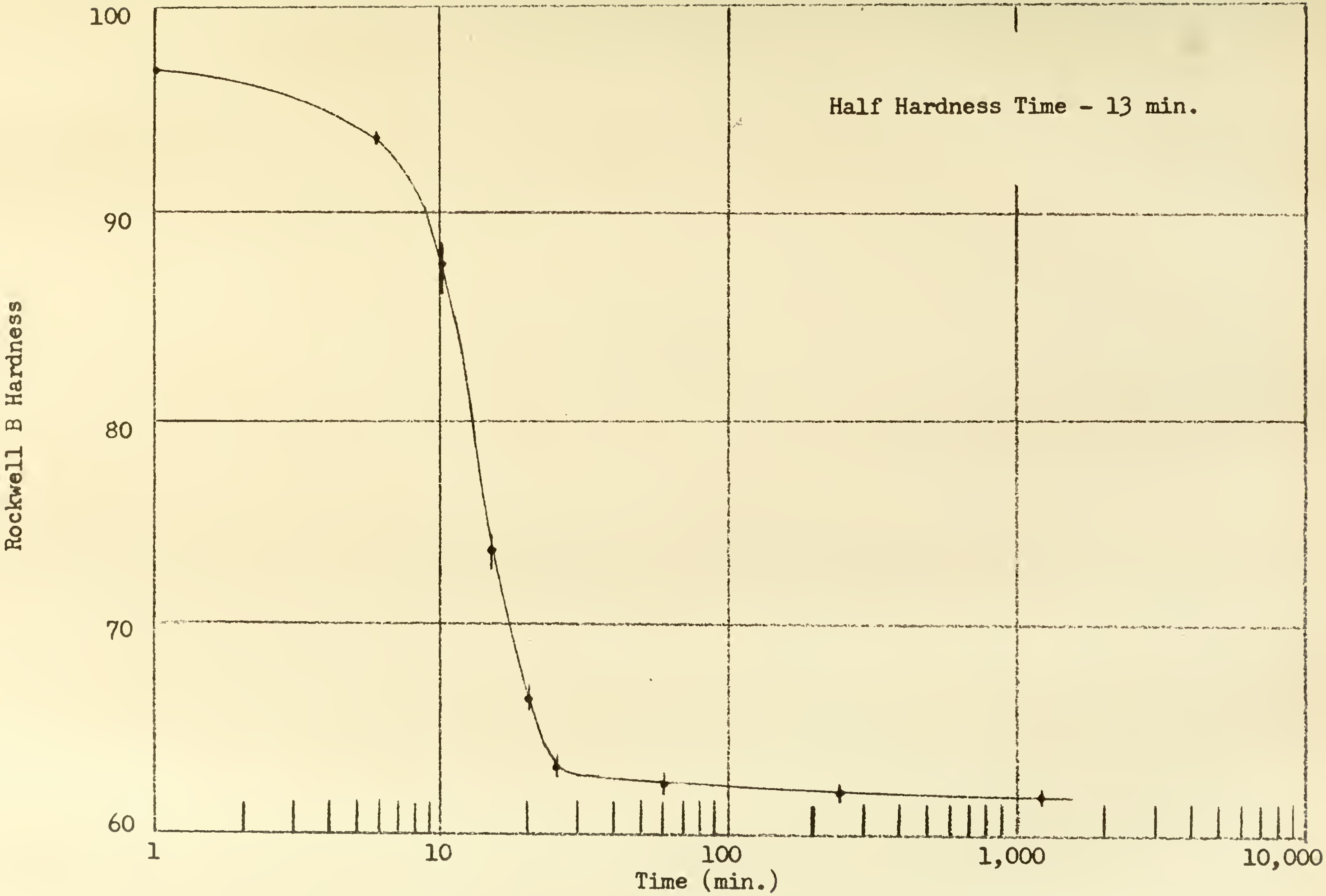


Fig. 11
Annealing Curve (580°C)

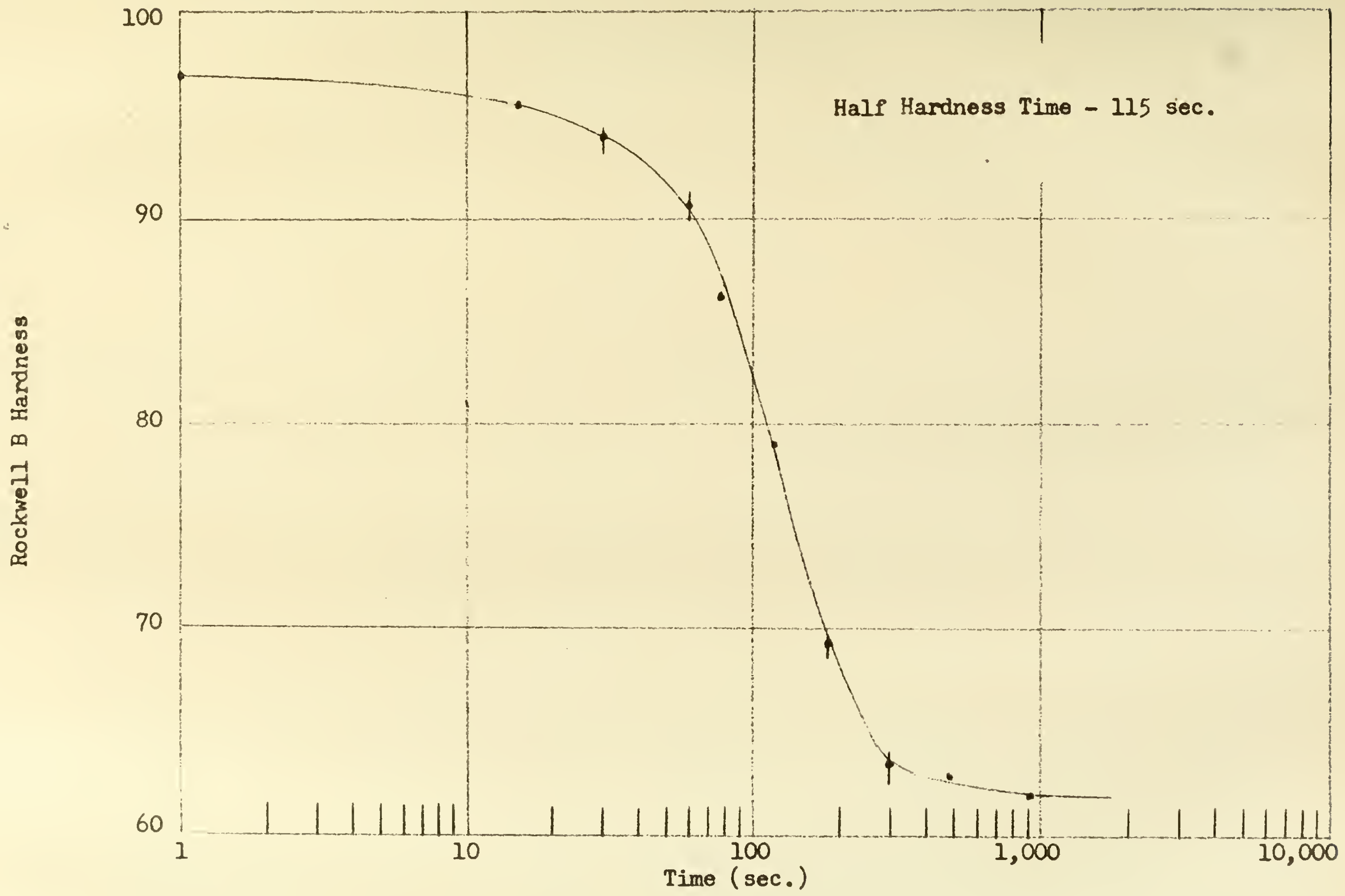


Fig. 12
Annealing Curve (605°C)

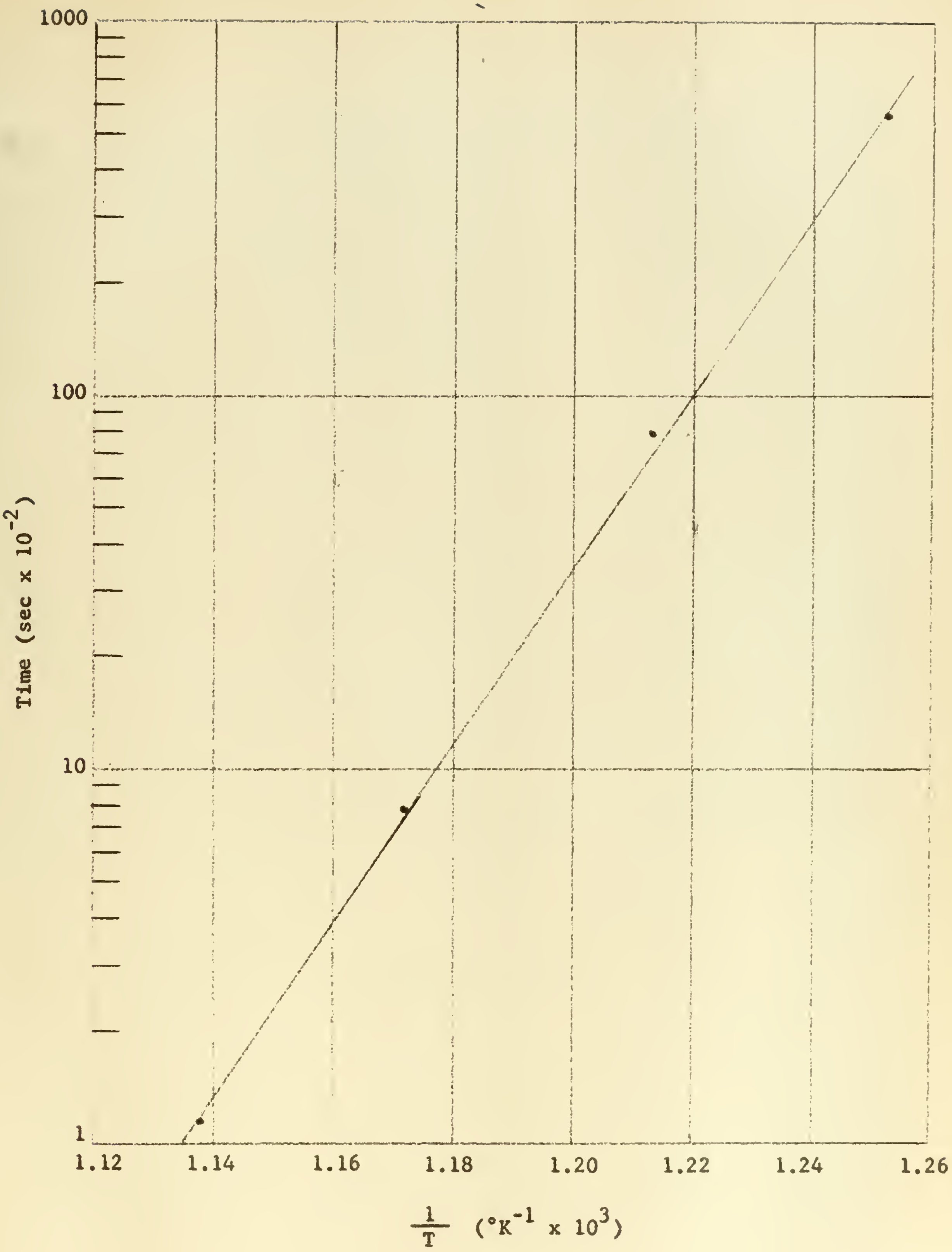


Fig. 13
Log Hardness Time vs $\frac{1}{T}$

EXPERIMENTAL PROCEDURE FOR EFFECT OF STRESS

A creep testing machine was used to examine the recrystallization of specimens under stress. Fig. 14 is a view of the experimental apparatus. It was decided to use a salt furnace rather than an air furnace because of the greater ease in maintaining a uniform temperature along the specimen length, because of the shorter time in reaching equilibrium temperature, and because of lessening of corrosion of the specimen during test. Houghton Liquid Heat 235 was again the salt used. In order to maintain an even temperature in the bath it was found necessary to use a stirring device.

The specimens employed in the test were as shown in Fig. 15. A tapered shape was decided upon because it made possible a varying stress with a single load on one specimen. The double taper makes possible a duplication of results in one specimen.

Timoshenko and Goodier[17] give an expression which allows calculation of the stress for a wedge shape due to a concentrated force at its apex.

$$S_r = \frac{P \cos \theta}{r(\alpha + \frac{1}{2} \sin 2\alpha)}$$

where S_r is the radial stress at radius r in p.s.i.; P is the load in pounds; r is the radius or distance from the apex of the wedge in inches; α is one-half the angle subtended at the apex of the wedge; and θ is the angle between the centerline of the wedge and the radial line to the point being considered. This

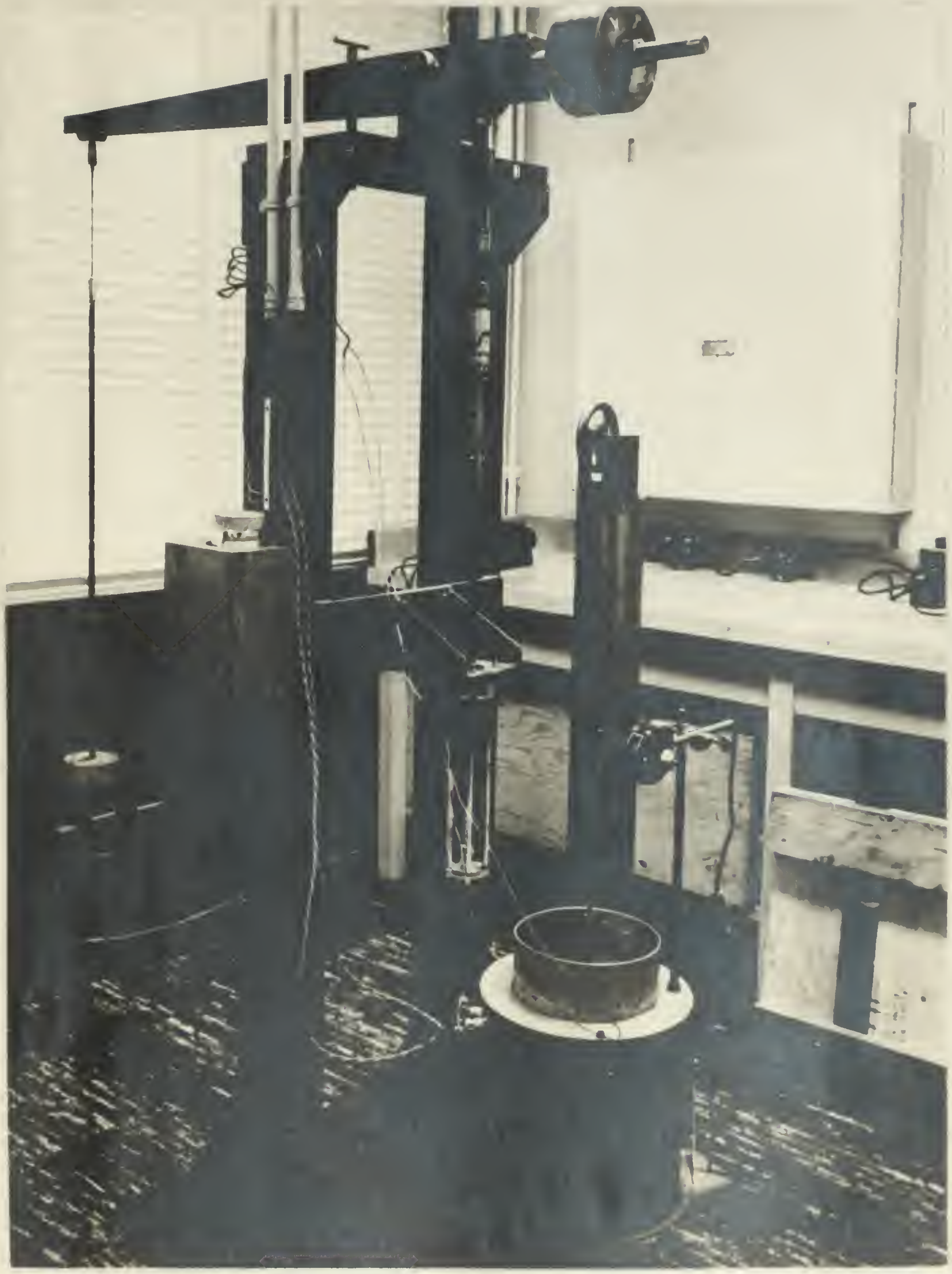


Fig. 14

Experimental Apparatus Showing Creep Machine and Salt Furnace

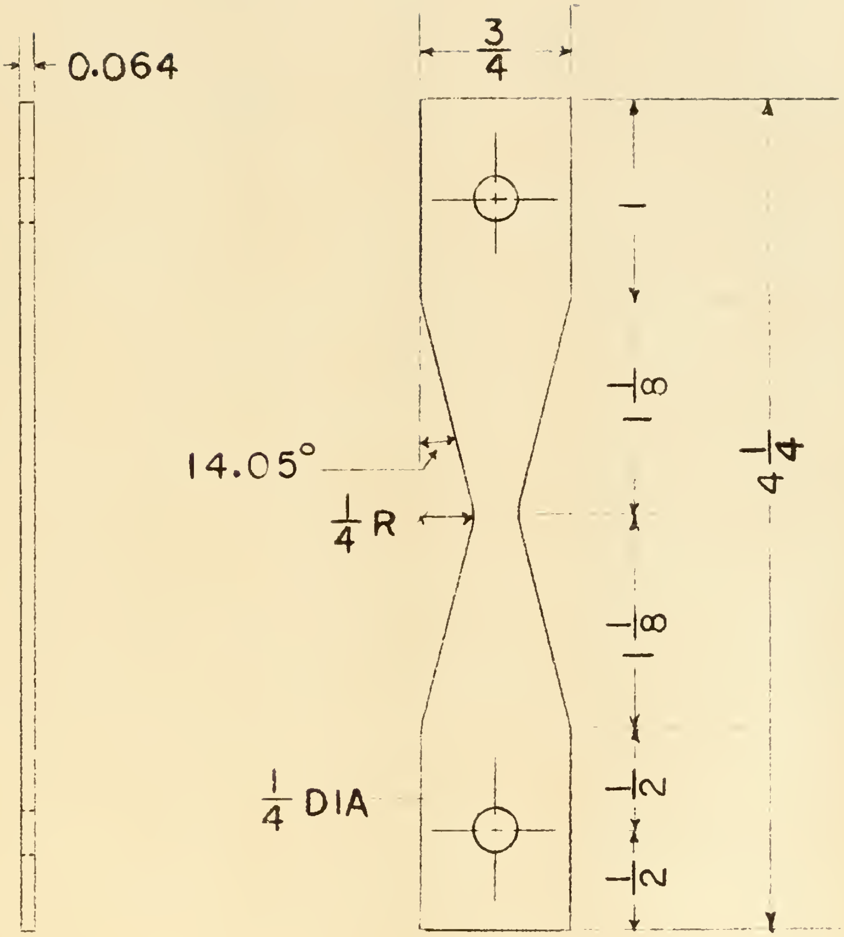


Fig. 15
Tapered Specimen Used for Stress Study

expression applies for a thickness of one inch. α for the specimens was 14.05 deg. θ was zero because all hardness readings were taken along the centerline. The region near the center of the specimens may be thought of as an overlap of wedges for which this formula does not apply.

Appendix II shows a sample calculation of stresses on a tapered specimen.

Temperatures were measured with a Chromel-Alumel thermocouple which was placed in a thin walled stainless steel tube to prevent shorting the leads. Aluminum shot was placed between the stainless steel wall and the thermocouple bead to improve heat transfer. This thermocouple was compared with an unshielded thermocouple sealed with Sauereisen refractory cement. No difference in temperature reading was observed. The cement sealed thermocouple was not used in the test because the cement would break down under action of the salt after a short period of time.

Small control specimens similar to the specimens used for the static recrystallization study were suspended in the salt bath near the tapered specimens in order to follow the recrystallization process and to serve as a guide for removal of the tapered specimens.

To overcome the uncertainty in measurement of time at temperatures where recrystallization is rapid, which would be particularly important if stress had a small effect, it was decided to work at temperatures where recrystallization proceeds slowly. Furthermore it was felt that if stress had an effect, this effect would be exaggerated if the stress acted for a longer time before the onset of

recrystallization.

RESULTS OF STRESS STUDY

Tables II and III show the conditions and results of the test. Fig. 16 shows the position of the stations referred to in Table III.

From Table II it can be seen that there was a slight permanent strain in all specimens. It was the purpose of the experiment to study the effects of stress below the plastic region, but it was felt that the small strain observed was confined to the center portion of the specimen, and it was desired to obtain as high a stress as possible within the elastic region.

For specimen No. 1 a temperature was chosen at which recrystallization would proceed at a slow rate. Examination of the control specimen facilitated an estimation of the time when recrystallization was just beginning. The test was interrupted at this time to determine if stress had any accelerating effect on initiating recrystallization. None was observed.

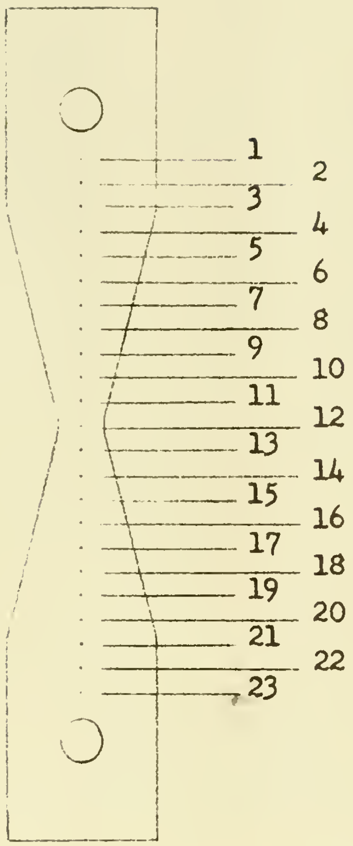
It was felt that since hardness readings change most rapidly in the steep portion of the sigmoidal annealing curve, any effect due to stress would be most readily discerned by an examination of a specimen that had just reached this condition. For this reason the test on specimen No. 2 was stopped near this region to note any effects. There were no differences in hardness along the specimen length that cannot be explained by a normal spread in readings.

The test on specimen No. 3 was very similar to that on specimen No. 2. A verification of results was desired. Again no significant difference in hardness readings along the length of the specimen was observed.

TABLE II

CONDITIONS FOR TEST SPECIMENS

Specimen No.	Stress Minimum Cross Section (psi)	Stress Maximum Cross Section (psi)	Bath Temperature (Deg. C)	Time Under Stress	Total Specimen Elongation (in.)
1	13550	3380	515	$23^{\underline{h}}$ $50^{\underline{m}}$	1/64
2	13570	3390	530	$15^{\underline{h}}$	1/64
3	13570	3390	529	$16^{\underline{h}}$ $50^{\underline{m}}$	1/64
4	13570	3390	512	$158^{\underline{h}}$	1/32



$\frac{1}{8}$ inch between readings

Fig. 16
Station Numbers for Table III

TABLE III

ROCKWELL B HARDNESS READINGS ON TAPERED SPECIMENS

Station	Specimen No. 1	Specimen No. 2	Specimen No. 3	Specimen No. 4
1	91	75	76.5	93
2	91	75.5	77.5	93.5
3	90.5	77	78.5	92.5
4	90.5	77	78.5	93
5	90.5	78	78	93
6	90	76.5	78	93
7	91	76.5	78	93
8	90.5	77	78	92.5
9	89.5	77	78	92.5
10	89	76.5	78	91
11	88	76.5	78	86.5
12	89	76	77.5	87
13	89	75.5	77.5	88.5
14	89.5	76.5	78	89.5
15	88.5	76	77	91.5
16	89	76	76.5	91.5
17	90.5	76	76.5	91.5
18	90.5	76	76.5	92
19	89.5	74	77	93
20	89.5	76	76.5	92
21	90.5	77.5	77	92
22	91	75	76	91.5
23	90.5	76.5	76.5	92

It was next desired to study the effect at a temperature where static recrystallization would not occur during the test time. Specimen No. 4 was under test for a week. There was a slight softening effect near the center of the specimen indicating an acceleration of recrystallization in that area.

Because of the longer period of time under test, specimen No. 4 showed the greatest permanent set due to creep and it was felt that the increased softening at the center was due to strain and not to stress. This effect of strain would tend to be in agreement with the results of Grant and Bucklin.[1]

SUMMARY OF RESULTS AND CONCLUSIONS

1. The activation energy of recrystallization for the material under investigation was determined to be 108,000 cal/gm-mol.

2. Within the limitations of the experimental technique employed there is no effect of elastic stress on recrystallization.

3. There appears to be an effect of plastic strain on recrystallization, but no quantitative results have been obtained.

A possible explanation of why strain causes an acceleration of recrystallization and stress does not is as follows. Stressing results in only a stretching of the bonds between atoms. Strain however is the result of a relative motion of atoms along slip planes. This relative motion may be the mechanism which facilitates the overcoming of the energy barrier by the atoms and the attainment of a lower energy state, for the process of recrystallization involves a movement of atoms as the interface of newly nucleated grains consumes the deformed matrix.

ACKNOWLEDGEMENTS

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APPENDIX I

CALCULATION OF RECRYSTALLIZATION ACTIVATION ENERGY

Figs. 9 through 12 show sigmoidal recrystallization curves for temperatures 525°C, 552°C, 580°C, and 605°C. Rockwell B hardness is plotted versus time. Fig. 13 is a plot of the logarithm of the time to reach half hardness versus the reciprocal of absolute temperature. The slope of the resulting straight line is a measure of the activation energy of recrystallization.

Using the Arrhenius equation

$$r = r_o \exp(-Q/RT)$$

This can be written

$$1/t = A \exp(-Q/RT)$$

where A is a constant and t is the time to reach a given percent recrystallization.

Taking the logarithm,

$$\ln 1/t = \ln A - Q/RT$$

or

$$\ln t = B + (Q/R)(1/T)$$

where B is a constant.

The latter equation is the slope intercept form for a straight line as it applies to the plot of Fig. 13. Using the method of least squares the equation of the line in Fig. 13 was found to be

$$\ln t = 57.2 + 54500\,(1/T)$$

Thus

$$Q/R = 54500$$

and

$$Q = 108{,}000 \text{ cal/gm-mol}$$

APPENDIX II

CALCULATION OF STRESSES ON TAPERED SPECIMENS

The formula for stresses in a wedge shape is

$$S_r = \frac{P \cos \Theta}{r(\alpha + \frac{1}{2} \sin 2\alpha)}$$

Since the value of stress is wanted only along the centerline, take Θ as zero. For the specimen used, $\alpha = 14.05^\circ = .2455$ rad. The formula is for unit thickness. Applying this formula to specimen No. 1, values are:

Thickness:	.06295 in.
Weight applied:	15.04 pounds
Lever arm:	10.20 to 1

$$S_r = \frac{(15.04)(10.2)}{r(.2455 + \frac{1}{2} \sin 28.1)(.06295)}$$

$$S_r = \frac{5080}{r}$$

At the minimum cross section

$$r = .375 \text{ in.}$$

$$S_r = \frac{5080}{.375} = 13550 \text{ psi}$$

At the maximum cross section

$$r = .375 + 1.125 = 1.500 \text{ in.}$$

$$S_r = \frac{5080}{1.500} = 3380 \text{ psi}$$

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